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ABSTRACT

A new design of a torsion balance for study of adhesion of ceramics is discussed. A torsion wire and a linear variable differential transformer are used as the sensing mechanism to monitor load and to measure pull-off force (adhesion force). The results of the investigation suggest that this torsion balance has proven to be valuable in studying the interfacial properties of ceramics in controlled environments such as in ultra high vacuum. The pull-off forces measured in dry, moist, and saturated nitrogen atmosphere demonstrate that the adhesion of silicon nitride to silicon nitride contacts remains low at humidities below 80 percent but rises rapidly above 80 percent. The adhesion at saturation is 10 times or more greater than that below 80 percent relative humidity. The adhesion in a saturated atmosphere arises primarily from the surface tension effects of a thin film of water adsorbed on the surfaces. The surface tension of the water film was calculated as 58×10^{-5} to 65×10^{-5} N/cm; the accepted value for water is 72.7×10^{-5} N/cm. Adhesion characteristics of silicon nitride in contact with metals, like the friction characteristics of silicon carbide to metal contacts, can be related to the relative chemical activity of metals in ultra high vacuum. The more active the metal, the higher is the adhesion.

INTRODUCTION

A crucial factor in the structural performance of ceramics, ceramic matrix composites, and ceramic coatings for use as components of advanced propulsion systems such as gas turbines and adiabatic diesel engines is the joining of ceramics to metals, the fiber/matrix bonding, or the coating/substrate bonding. Adhesion is developed in joining, bonding, and film formation processes. Adhesion also occurs in friction and wear processes of ceramics resulting in high friction and heavy surface damage. This is of importance in determining the high-temperature phenomena and the life of ceramic components. Therefore, adhesion of ceramic surfaces at high temperatures is of interest to those concerned with materials and process technology as well as the understanding of key phenomena of ceramics (References 1 to 7).

Adhesion has been extensively studied for metals and polymers, but only recently examined for ceramics (References 8 to 18). There is far less understanding of the forces and bonding if two pieces of ceramics are brought into contact, especially in the case of high-temperature phenomena.

Adhesion is a manifestation of mechanical strength over an appreciable area and is due to many factors including chemical bonding and the fracture processes involved in failure of the interface. The strength of adhesion is expressed as the force to pull the surfaces apart and is called the pull-off force to distinguish it from thermodynamic adhesion and from surface forces (Reference 19). The pull-off force undoubtedly depends on the area of real contact, the interfacial bond strength, the micromechanical properties of the interface, and modes of junction rupture. Unfortunately, there is no satisfactory theory or experimental method for determining the area of real contact. Factors which influence the adhesion and deformation behavior of ceramics such as vibration and environment also need to be considered. The problem of vibration may cause junction growth in the contact zone. There are many unknown and unresolved problems, and adhesion studies of ceramics are best performed only through refined experiments under carefully controlled laboratory conditions, such as in a high vacuum or in an inert gas in order to avoid secondary effects.

This paper attempts to describe an adhesion measuring apparatus, which allows measurements in ultra high vacuum even at high temperatures up to 1200 °C, developed by the present authors. The materials which have been considered are ceramic and fiber materials such as silicon nitride and silicon carbide. In this paper, however, adhesion of silicon nitride will be discussed.

The apparatus was adapted for this purpose from the Cavendish balance used in measuring gravitational forces in 1798 and also from a similar balance invented by Coulomb in 1784 to 1785 for studying forces of electrical attraction and repulsion (References 20 and 21). After a description of the principles of this method, the adhesion apparatuses, and experimental procedures, some results obtained from pretests were first described and are used to evaluate the performance of the torsion balance. In the pretests forces of magnetic attraction were examined using the torsion balance. Two types of adhesion experiments were conducted to examine the adhesion behavior of silicon nitride. The first is conducted with silicon nitride in contact with itself in dry, moist, and saturated nitrogen and the second is conducted primarily with silicon nitride in contact with transition metals in vacuum.

THE PRINCIPLE AND ANALYSIS OF SPRING STIFFNESS

The Principle of Operation

The adhesion measuring apparatus used in this investigation is a torsion balance adapted from the Cavendish balance. The torsion balance consists of a mass m and a sensor, such as an electromechanical transducer, mounted at opposite ends of a horizontal rod which is supported at its center by a vertical wire, such as a music wire (Figure 1). Another mass M is brought up to the position shown. In order to use the balance, the mass M is moved toward the mass m, presses against it and twists the wire through a small angle with a normal force, the normal loading process, thereby moving the sensor. The mass M is then gradually moved horizontally backward until the two masses m and M are pulled apart in a normal direction, the unloading process. If the force of adhesion between the two masses m and M is zero, the mass M separates from m at its original position and untwists the wire, thereby moving the sensor back to its original position. If an adhesive force between the two masses m and M is present, the force twists the wire as mass M moves backward until the wire develops sufficient force to separate the surfaces of masses m and M in the normal direction.

In this system, the attractive force of adhesion and the force required to pull the surfaces of the two masses apart, the pull-off force, act along a horizontal direction without the effect of gravity. The weights of all the components (such as rod, sensor, wire, etc.) act in the vertical direction due to gravity.

By using a fine music wire in the practical adhesion apparatus, the displacement of the mass m may be made sufficiently large so that the adhesion, that is, pull-off force, can be measured accurately.

Torsional Spring Stiffness of Wire

In order to find the type of motion performed by the torsion balance after the mass has been displaced from its neutral position and to determine the value of torsional spring stiffness of the wire used in the torsion balance, a simple spring-mass system, a torsional pendulum, shown in Figure 2 was considered (References 22 and 23). The torsional pendulum is suspended vertically with the wire. Figure 2(a) presents the side view of the system consisting of a mass with moment of inertia J about the wire axis, suspended by the wire of stiffness K. Figure 2(b) presents an end view. In other words, an irregular body is simply attached to the spring wire in such a way that the mass is forced to rotate about the axis of the wire. The system is also used to determine experimentally the stiffness K of the spring materials, that is, the spring constant k of wires used in this study.

It is assumed in the following formulas that a wire spring is in no case stressed beyond the elastic limit (i.e., that it is perfectly elastic) and that it is subject to Hooke's law. All external torques acting on the suspended mass are considered positive clockwise when viewed from below (see Figure 2).

A free-body diagram of the mass with all the torque acting on it is shown in Figure 2(c). Because of the twist θ of the wire, a counterclockwise torque $K\theta$ is exerted, and Hooke's law gives $q = -K\theta$. For convenience a torque is usually drawn in such a direction that minus signs are avoided, and the free-body diagram of figure 2(c) is obtained. For a solid circular wire of diameter d and length L, the torque q is represented by the formula

$$q = \frac{-\theta G I_p}{L} \tag{1}$$

The torsional stiffness K is then given by the formula

$$K = \frac{GI_p}{L} = \frac{\pi G d^4}{32L} \tag{2}$$

Applying Newton's law gives $-K\Theta = J\ddot{\Theta}$, or

$$J\ddot{\Theta} + K\Theta = 0 \tag{3}$$

which is the differential equation of the harmonic oscillator. Introducing the quantity

$$\omega_n = \sqrt{\frac{K}{J}} \tag{4}$$

in Equation (3) puts it in the following form:

$$\ddot{\Theta} + \omega_n^2\Theta = 0 \tag{5}$$

The solution is given as

$$\Theta = A\cos(\omega_n t - \gamma) \tag{6}$$

where t is the time and ω_n is the natural angular frequency which is 2π times the natural frequency f_n. The constants A and γ are determined by the way in which the motion was started. The motion in this case is harmonic with the constant A, the arbitrary amplitude, and the constant γ, the arbitrary phase angle between the motion and the reference motion $\cos \omega_n t$.

The natural frequency f_n of the system is then given by the expression

$$f_n = \frac{1}{2\pi}\sqrt{\frac{K}{J}} \tag{7}$$

The natural period of the motion is therefore

$$\tau_n = 2\pi\sqrt{\frac{J}{K}} \tag{8}$$

It is to be noted from Equation (8) that the natural period of the vibration decreases with the spring stiffness K, which is a measure of the restoring force, increases with an increase of mass m (i.e., with moment of inertia J), and is wholly independent of the arbitrary constants A and γ.

If additional irregular bodies (masses) having the moment of the inertia ΔJ are simply attached to the slender wire in such a way that the axis of one of their principle moments of inertia coincides with the axis of the wire, the total moment of inertia about the suspension axis is given as $J + \Delta J$ (Figure 2(d)). From Equation (8), the natural period of the oscillation τ_{n1} for $J + \Delta J$ would be

$$\tau_{n1} = 2\pi\sqrt{\frac{J + \Delta J}{K}} \tag{9}$$

By measurement of the natural periods τ_n and τ_{n1} of the oscillations, the value K of the torsional stiffness is then obtained from Equations (8) and (9) in the form

$$K = \frac{4\pi^2\Delta J}{\tau_{n1}^2 - \tau_n^2} \tag{10}$$

This is a convenient form in which to determine experimentally the torsional stiffness K of a wire.

The torsional spring stiffness of wire can be determined experimentally also by the torsional moment-deflection relationship $q = -K\Theta$ for a slender wire (Figure 3). In Figure 3 is shown a view of the pendulum consisting of the wire and rod held horizontally. The weight ΔMg is attached to the rod at a distance a from the wire. The torque q is given as $q = \Delta Mg\, a$ (Figure 3(b)), and the torsional stiffness of wire is given by

$$K = \frac{\Delta M g\ a}{\theta} \qquad (11)$$

Thus, the stiffness K of the spring materials can be determined experimentally using Equation (2), (10) or (11).

MATERIALS

Hot pressed polycrystalline magnesia doped silicon nitride and sintered polycrystalline magnesia-partially stabilized zirconia (Mg-PSZ) were used in the adhesion experiments (References 24 and 25).

All metals used in the experiments were also polycrystalline. The titanium was 99.97 percent pure, and all the other metals were 99.99 percent pure.

APPARATUSES

Two apparatuses used in this investigation were based on the torsion mechanism. One was used for the pretests in carefully controlled laboratory air. The second apparatus was used for the adhesion experiments of the ceramics and was placed in a vacuum chamber (Figure 4). They were basically a pin on a flat configuration, as typically shown in Figure 4. The flat (or the pin) was mounted on a support and retained on a micrometer-head-screw-driven platform moved by an electric motor. The pin (or flat) was mounted on one end of a movable beam. A free-moving, rod-shaped magnetic core was mounted on the other end of the beam. The coils of a linear variable differential transformer were mounted on a stationary beam. There was no physical contact between the movable magnetic core and the coil structure. The movable beam was supported by a music wire acting as a torsion spring.

The flat (or the pin) specimen was moved toward the pin (or the flat), pressed against it with a known force, and then moved back horizontally until the pin and flat were pulled apart. The displacement of the moving pin (or the flat) was continuously monitored using the linear variable differential transformer during experiments. The displacement of the pin (or the flat) provided a measure of the applied normal load or pull-off force, adhesion.

EXPERIMENTAL PROCEDURES

Specimen Preparation

The contacting surfaces of the ceramic specimens were hemispherical or flat and were polished with diamond powder 3 μm and 1 μm in diameter. The polished faces had smooth, bright, lustrous surfaces. The radius of curvature of the hemispherical silicon nitride pins were 1.6 mm.

The contacting surfaces of the metals were hemispherical and were polished first with diamond powder 3 μm and 1 μm in diameter and then with aluminum oxide powder 1μm in diameter. The radii of curvature of the metal pins were 0.79 or 1.6 mm.

All the specimens used in this investigation were rinsed with absolute ethanol before the experiments.

Procedures

The pretests and two types of adhesion measurements for silicon nitride were conducted.

As pointed out previously, the stiffness K of the music wires can be determined by Equation (2), (10), or (11). The results are presented in Table 1. Using the values of the stiffness K obtained from Equation (10), the torsion balances were calibrated to measure force and were used to determine unknown forces acting on the torsion wires. The force acting on the torsion wire is directly proportional to the deflection of the movable beam, as monitored by the linear variable differential transformer.

Pretests - The pretests were conducted in laboratory air with hemispherical ferrite pins (NiO, 11.0; ZnO, 22.2; Fe_2O_3, 66.0 at %) in contact with flat surfaces of microscope slides made from high purity glass. A permanent magnet (magnetic flux density, 1200 G) was mounted behind a stack of glass flats and retained in a vice mounted on a micrometer-head-screw driven platform moved by an electric motor (Figure 5). The thicknesses of the glass flat stacks were 5.71 to 7.84 mm. The ferrite pin was mounted on one end of the movable beam.

The magnetic induction or magnetic flux density B, which the permanent magnet produced at the site of contact between the ferrite pin and glass flat, was also measured with a conventional Gauss-meter sensor in contact with the surface glass flat.

For pull-off force measurements, the pin specimens were brought into contact with the flat specimens by moving the micrometer-head-screw forward at speeds of 3.7, 1.8, or 1.2 mm/s. Contact was maintained for 20 to 30 sec and then the pin and flat specimen surfaces were pulled apart by moving the micrometer-head-screw backward. The displacement of the pin was monitored by a linear variable differential transformer.

Adhesion Measurements - The first set of experiments was conducted in dry, moist, and saturated nitrogen atmosphere. The entire torsion balance was placed in the chamber presented in Figure 4.

After dry nitrogen was admitted into the system, the silicon nitride pin and flat specimens were placed in the experimental apparatus and maintained in dry nitrogen for 15 min. Adhesion measurements were then conducted in dry nitrogen. Further, the atmosphere was humidified to the desired relative humidity by admitting humid nitrogen into the system and adhesion measurements were conducted in the humid nitrogen.

The second set of experiments was conducted in vacuum. The pin and flat specimens were placed in a vacuum chamber, and the system was evacuated and baked out to achieve a pressure of 30 nPa (Figure 4). In-situ adhesion experiments were first conducted with the as-received specimens in a 30 nPa vacuum. Further, ion-sputter etching of the pin and flat specimens was performed with a beam energy of 3000 eV at 20 mA beam current with an argon pressure of 0.7 mPa. The ion beam was continuously rastered over the specimen surface. After sputter etching, the system was reevacuated to a pressure of 30 nPa or lower and then in-situ adhesion measurements were conducted with the ion-sputter cleaned specimens in a 30 nPa vacuum. The surface cleanliness of the pin and flat specimens was verified by x-ray photoelectron spectroscopy analysis.

For adhesion measurements both in nitrogen and in vacuum, the pin specimen was brought into contact with the flat specimen by moving the micrometer-head-screw forward at a speed of 0.30 mm/min in a controlled nitrogen atmosphere. Contact was maintained for 30 sec and the pin and flat specimen surfaces were pulled apart by moving the micrometer-head-screw backward. The displacement of the flat specimen was monitored by a linear variable differential transformer.

RESULTS AND DISCUSSION

Pretests

The magnetic field which the permanent magnet produced at the contact site between the ferrite pin and the glass flat was primarily responsible for the measured pull-off force (Figure 5.).

The magnetic field B at the distance t from the permanent magnet having magnetic moment M_1 is given by

$$B = \frac{M_1}{2\pi t^3} \tag{12}$$

from References 26 and 27. In the equation the magnetic field B is strongly related to the distance t.

Figure 6 presents data for magnetic flux densities B measured as a function of the distance t, which is the spacing between the surfaces of the permanent magnet and the Gaussmeter sensor. The data reveal a decrease in magnetic flux density with an increase in distance. The relation between the magnetic flux density B and the distance t is given by an expression of the form $B \propto t^{-3}$ from Figure 6. The -3 power is interpreted using Equation (12).

The force of magnetic attraction F acting on the ferrite pin due to the existence of the permanent magnet is theoretically given by (Reference 27)

$$F = \frac{3M_1M_2}{2\pi\mu_o t^4} \tag{13}$$

where

M_1 magnetic moment of the permanent magnet
M_2 magnetic moment of the ferrite pin
μ_o the permeability of vacuum
t the distance between the permanent magnet and the tip of the pin specimen

Further, the relationship between the attractive force F and the magnetic flux density B is theoretically given by

$$F = \frac{3M_2}{\mu_o} \left(\frac{2\pi}{M_1}\right)^{1/3} B^{4/3} \qquad (14)$$

Figures 7 and 8 present the pull-off forces necessary to separate the ferrite pin from the surface of the glass flats. When the ferrite pin detaches from the glass surface, the applied pull-off force is balanced by the magnetic attraction acting in the opposite direction. The pull-off forces of the ferrite-glass contacts shown in Figures 7 and 8 are due primarily to the magnetic attraction. The narrower the distance between the permanent magnet and the ferrite pin, the greater the pull-off force required (Figure 7). Equivalently the higher the magnetic flux density B, the greater the pull-off force (Figure 8). The relationships of the pull-off force to the distance t and to the magnetic flux density B are given by two expressions $F \propto t^{-4}$ and $F \propto B^{4/3}$, respectively, from Figures 7 and 8. The -4 power and the 4/3 power are interpreted using Equations (13) and (14), respectively. Note that there were no changes in pull-off force with contacting and separating speeds of 2.2, 1.1, and 0.72 mm/min.

Adhesion Measurements

Nitrogen Atmosphere - For a hemispherical silicon nitride pin in elastic contact with a silicon nitride flat in dry, moist, and saturated nitrogen, adhesion remained low at humidities below 80 percent but rose rapidly above 80 percent (Figure 9). Note that there was no change in adhesion with normal load in the range of 0.2 to 0.8 mN in dry, moist, and saturated nitrogen.

It is anticipated that the adhesion observed in a saturated nitrogen atmosphere arose primarily from the surface tension effects of a thin film of water adsorbed on the silicon nitride surfaces. The happenings at the interface may be visualized as follows: a silicon nitride pin is essentially in elastic contact with a silicon nitride flat surface, with a thin film of water between them (Figure 10(a)). When the normal load is removed and the elastic stresses within the bulk of the specimens are released, the interfacial junctions are broken one by one. When the pin (of radius R) detaches from the flat (Figure 10(b)), the applied separation force is balanced by the surface tension of a thin film of water resisting the extension of the surface. Suppose the liquid collects to form a pool at the tip of the hemispherical pin and the radius of curvature of the profile of the meniscus is r. If the meniscus is very small ($r \ll R$) and the liquid completely wets the surface (i.e., the contact angle is zero), the pressure p inside the liquid is less than atmospheric pressure by approximately T/r, where T is the surface tension of the liquid. This acts over an area πa^2 of the water pool, giving a total adhesive force of $p\pi a^2$ (i.e., $\pi a^2 T/r$). To a close approximation $a^2 = 2R \times 2r$, where R is the radius of curvature of the spherical surface. The resulting adhesive force is the following (see References 16 and 28):

$$Z = 4Rr\pi \left(\frac{T}{r}\right) = 4\pi RT \qquad (15)$$

Adhesion is thus independent both of the thickness of the water film and the applied normal load. The surface tension calculated from these results (Figure 9) is 58×10^{-5} to 65×10^{-5} N/cm. The accepted value for water is 72.7×10^{-5} N/cm. This discrepancy may be due to the surface roughness and irregularities of roundness or flatness of the silicon nitride specimens. The irregularities can affect the radii of curvature of the hemispherical pin and meniscus.

Vacuum - Adhesion experiments were conducted with silicon nitride in contact with metals and ceramics in vacuum. Typical results are presented in Figure 11. The marked difference in adhesion for the as-received and the ion-sputter cleaned specimens shows the effect of adsorbates on the adhesion properties. The pull-off forces for metals and ceramics contacting silicon nitride flats in the as-received condition were relatively small. However, removing adsorbed films from the surfaces of ceramics and metals resulted in strong interfacial adhesion, when two such solids were brought into contact.

Pauling, in the 1940's, formulated a resonating-valence-bond theory of metals and intermetallic compounds in which numerical values could be placed on the bonding character of the various transition elements (Reference 29). Since the d-valence bonds are not completely filled in transition metals, they are responsible for such physical and chemical properties as cohesive energy, shear modulus, and chemical stability. The greater the amount or percentage of d-bond character that a metal possesses, the less active is its surface. While there have been critics of this theory, it appears to be most plausible in explaining the interfacial interactions of transition metals in contact with ceramics as well as with themselves. It was found that the coefficient of friction for transition metals in contact with themselves or ceramic materials such as silicon carbide depends heavily on the d-bond character of the metal (References 8 and 30). These data indicate a decrease in coefficient of friction with an increase in d-bond character, as predicted from Pauling's theory.

The pull-off force of various metals in contact with the hot-pressed polycrystalline silicon nitride is presented in Figure 12 as a function of the d-bond character of the transition metal. The adhesion properties of metal-silicon nitride contacts, like the friction properties of metal-silicon carbide sliding contacts, are related to this character. The greater the percentage of d-bond character, the less active is the metal and the lower is the adhesion. Conversely, the more active the metal, the higher is the adhesion. Titanium, which is a chemically active metal, exhibits a considerably higher adhesion in contact with silicon nitride than does rhenium, which is a metal of lesser activity.

CONCLUDING REMARKS

Based upon the development of adhesion measuring equipment and fundamental studies of adhesion conducted with both nonoxide and oxide ceramics, the following remarks can be made.

This torsion balance, which allows adhesion measurements in vacuum, has proven to be valuable in studying the interfacial properties of ceramics and metals. The main advantages of this system are its simple, low cost, and accurate in-situ measurements. Forces as small as 1 μN can be measured.

The adhesion of silicon nitride to silicon nitride contacts measured in dry, moist, and saturated nitrogen atmosphere remains low at humidities below 80 percent but rises rapidly above 80 percent. The adhesion at saturation is 10 times or greater than that below 80 percent relative humidity. The adhesion in a saturated atmosphere arises primarily from the surface tension effects of a thin film of water adsorbed on the surfaces. The surface tension of the water film was calculated as 58×10^{-5} to 65×10^{-5} N/cm. The accepted value for water is 72.7×10^{-5} N/cm.

Adhesion characteristics of silicon nitride in contact with metals, like the friction characteristics of silicon carbide to metal contacts, can be related to the relative chemical activity of metals in ultrahigh vacuum. The more active the metal, the higher is the adhesion.

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NOTATION

d diameter of the wire

f_n natural frequency of the system

G shear modulus for wire material

K torsional stiffness of the wire (it takes the place of the spring constant k; the value of K is the moment (torque) necessary to twist the wire a unit angle (1 rad))

I_p polar moment of inertia of circular cross section

J moment of inertia of the torsionally vibrating mass about the wire axis

L length of the wire

q torque (i.e., torsional moment)

θ angle (i.e., the angular displacement of the mass from its equilibrium position)

$\dot{\theta}$ angular velocity

$\ddot{\theta}$ angular acceleration

τ_n natural period of the motion

ω_n natural angular frequency (or circular frequency), which is 2π times the natural frequency f_n

TABLE I. - TORSIONAL STIFFNESS K OF MUSIC WIRES

Equation	Pretests K, N/mm	Adhesion measurements K, N/mm
K obtained from Equation (2)	9.69	2.25
K obtained from Equation (10)	9.62	2.35
K obtained from Equation (11)	9.07[a]	2.45[a]
	8.90[b]	2.25[b]

[a]A wire was twisted in a direction of normal load to be applied during adhesion experiments.
[b]A wire was twisted in a direction of pull-off force to be applied during adhesion experiments.

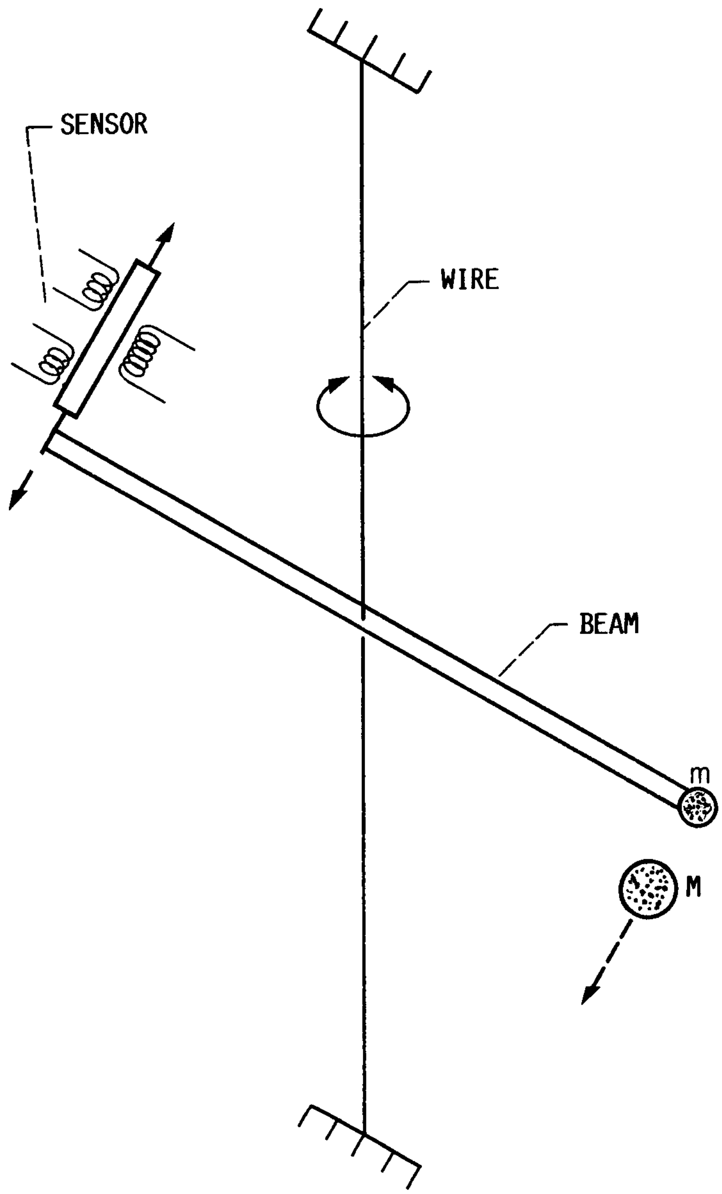
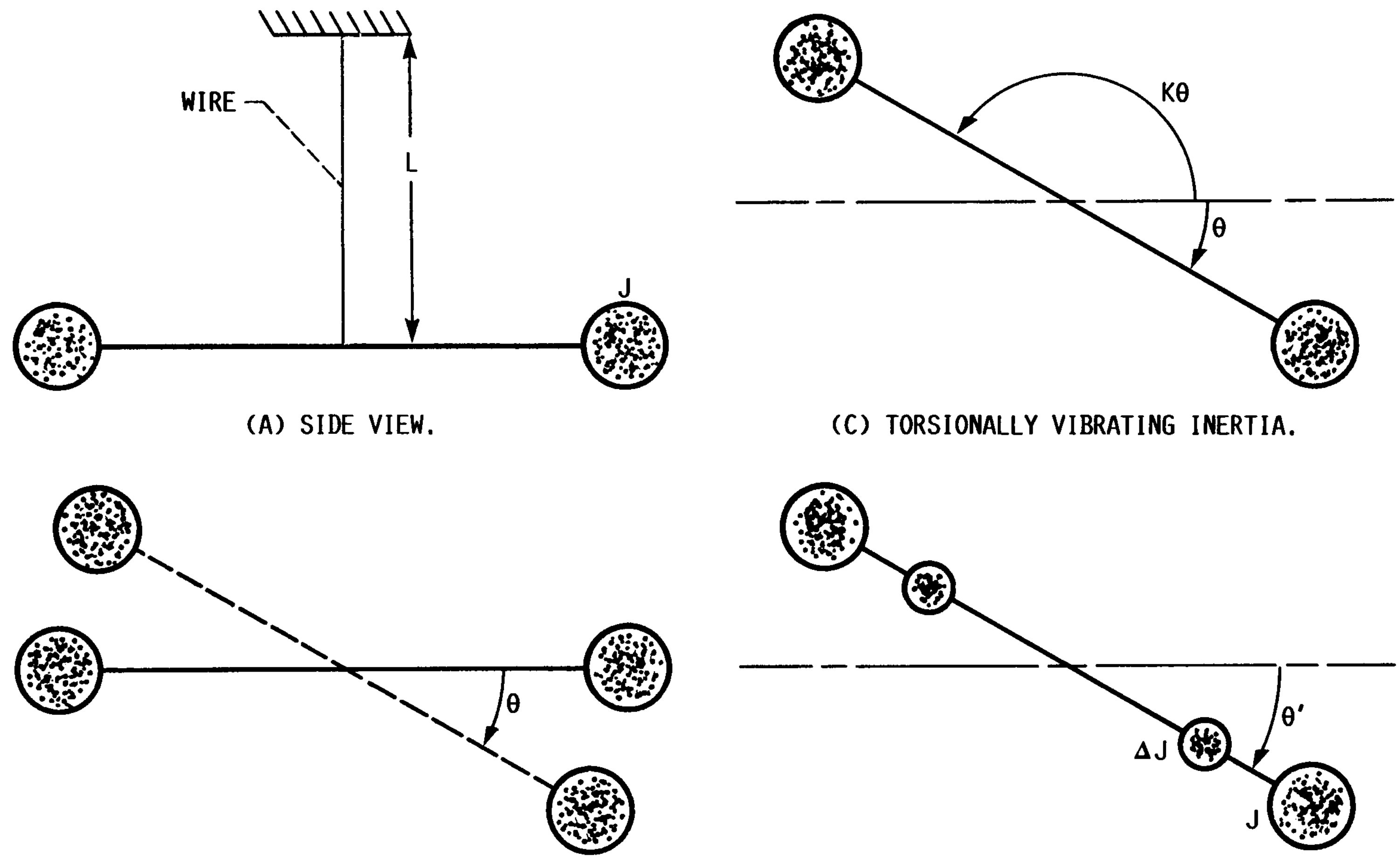


FIGURE 1. - SCHEMATIC DIAGRAM OF TORSION BALANCE IN PRINCIPLE ADAPTED FROM THE CAVENDISH BALANCE.



(B) END VIEW.

(D) $J + \Delta J$.

FIGURE 2. - TORSIONAL PENDULUM, CONSISTING OF CONCENTRATED INERTIA J AT THE END OF A MASSLESS WIRE OF STIFFNESS K.

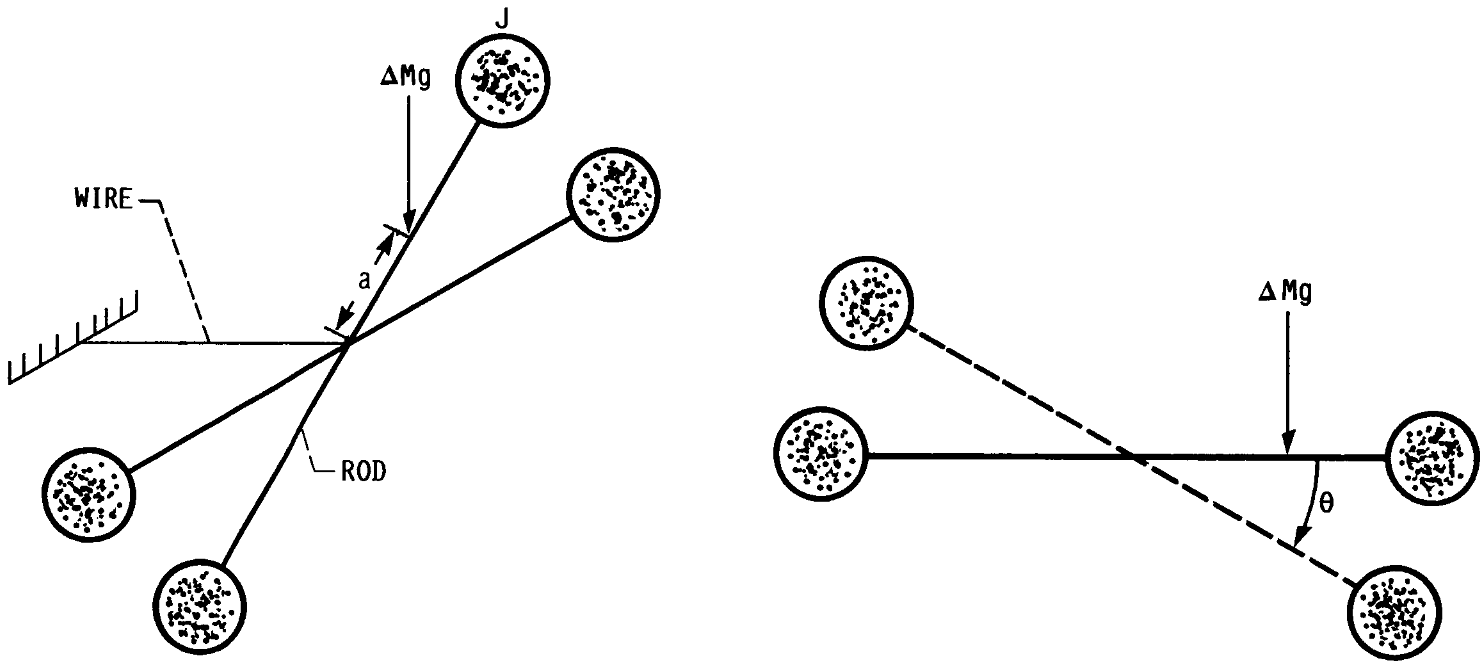


FIGURE 3. - TORSIONAL MOMENT ACTING ON A WIRE.

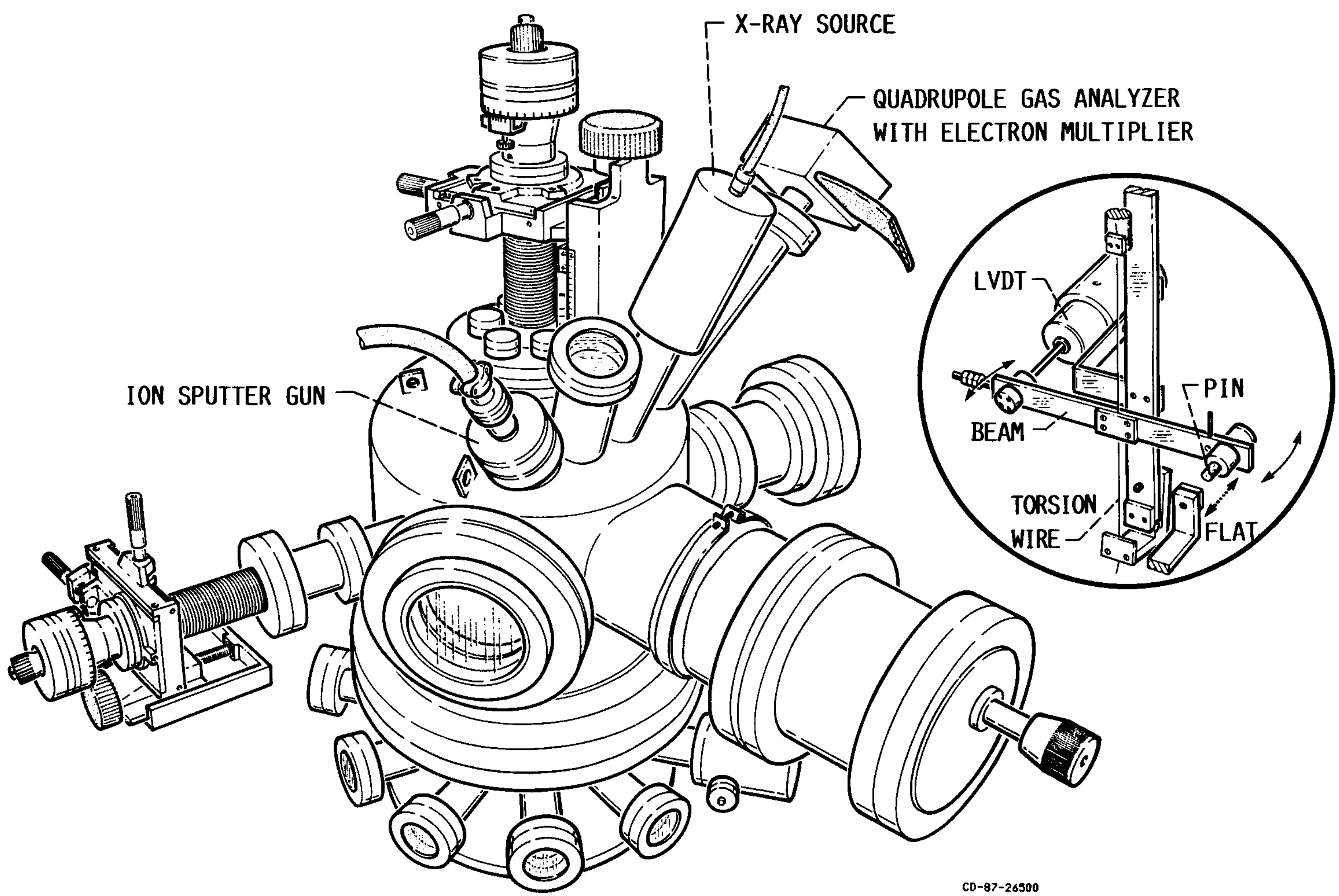


FIGURE 4. - APPARATUS FOR MEASURING ADHESION IN ULTRA HIGH VACUUM.

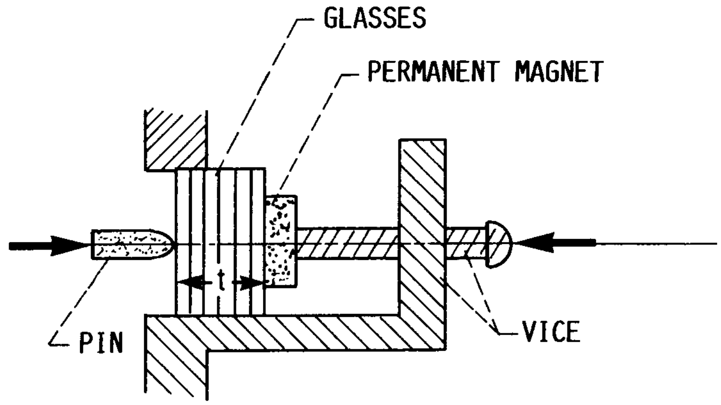


FIGURE 5. - SCHEMATIC CONFIGURATION OF A FERRITE PIN, GLASSES, AND A PERMANENT MAGNET (A FERRITE PIN UNDER THE ACTION OF TRANSLATIONAL FORCE IN A GRADIENT MAGNETIC FIELD PRODUCED BY A PERMANENT MAGNET).

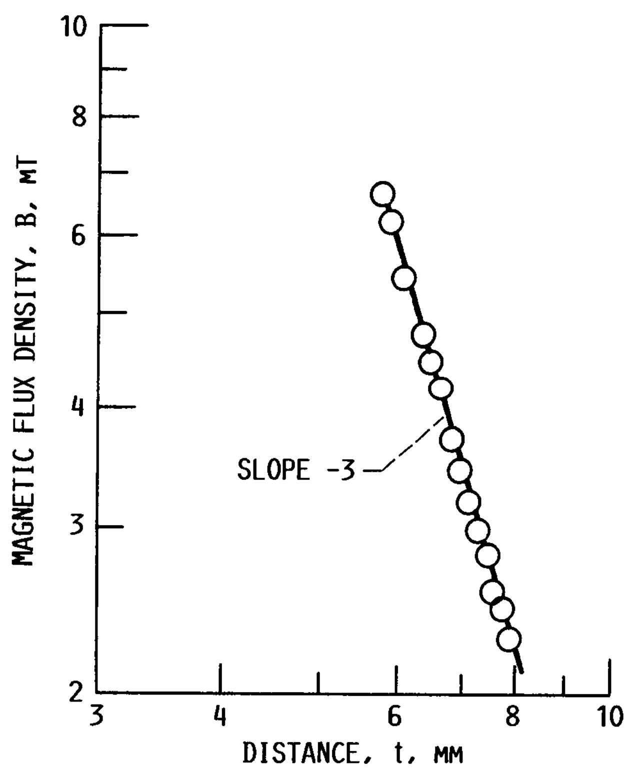


FIGURE 6. - MAGNETIC FLUX DENSITY AS A FUNCTION OF DISTANCE FROM A PERMANENT MAGNET IN LABORATORY AIR.

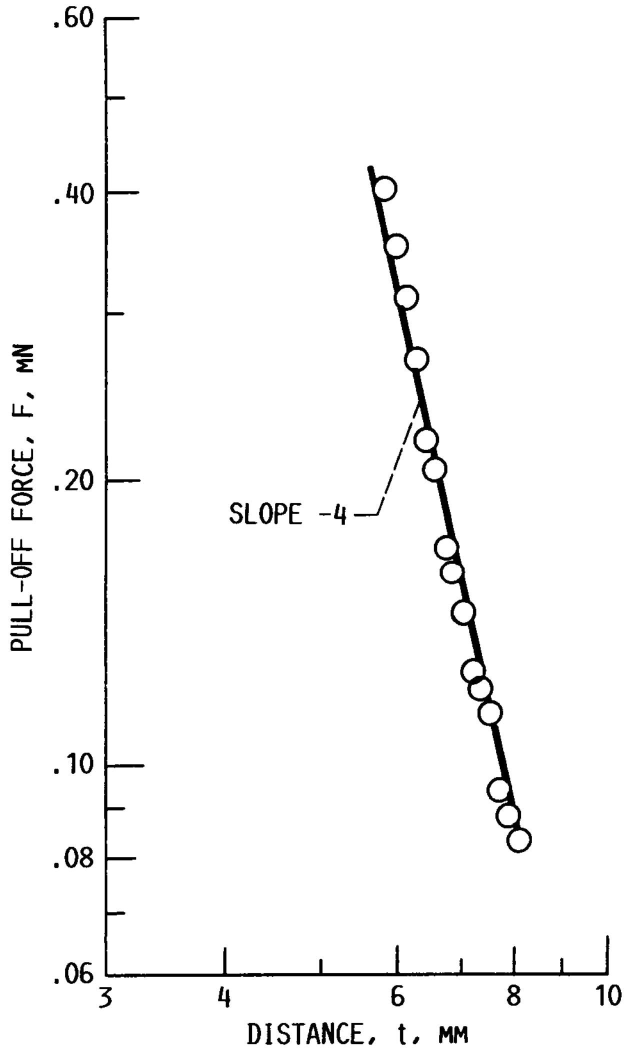


FIGURE 7. - PULL-OFF FORCES AS A FUNCTION OF DISTANCE BETWEEN A PERMANENT MAGNET AND A Ni-Zn FERRITE PIN.

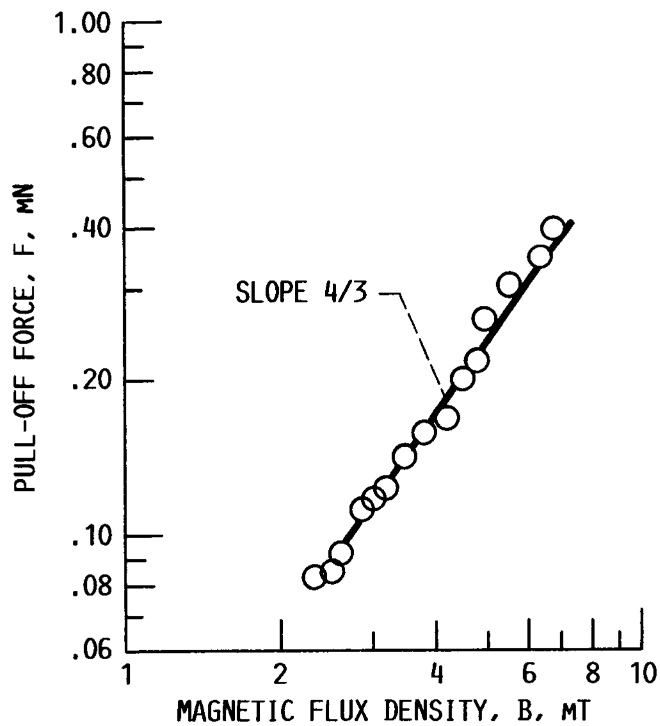


FIGURE 8. - PULL-OFF FORCES AS A FUNCTION OF MAGNETIC FLUX DENSITY AT AN INTERFACE BETWEEN A FERRITE PIN AND A GLASS FLAT.

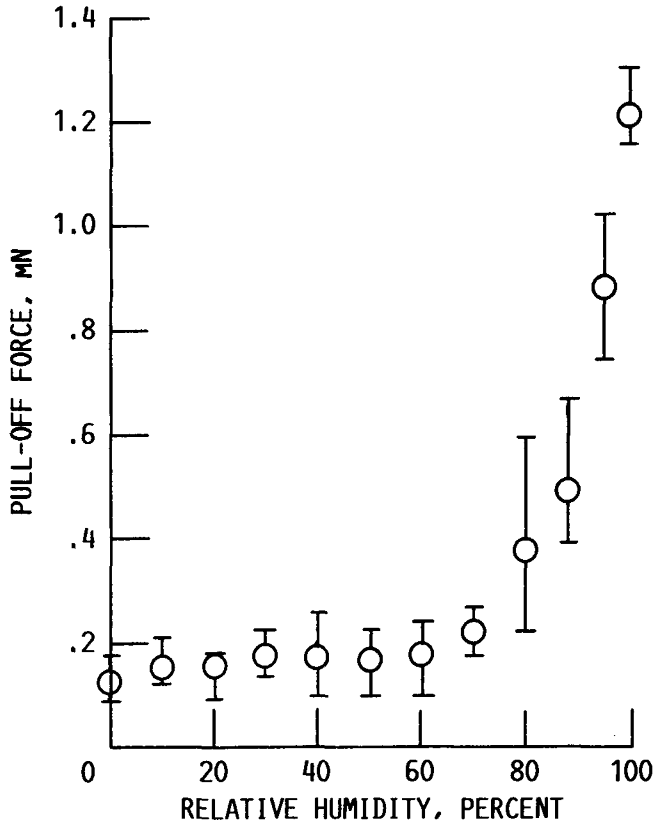
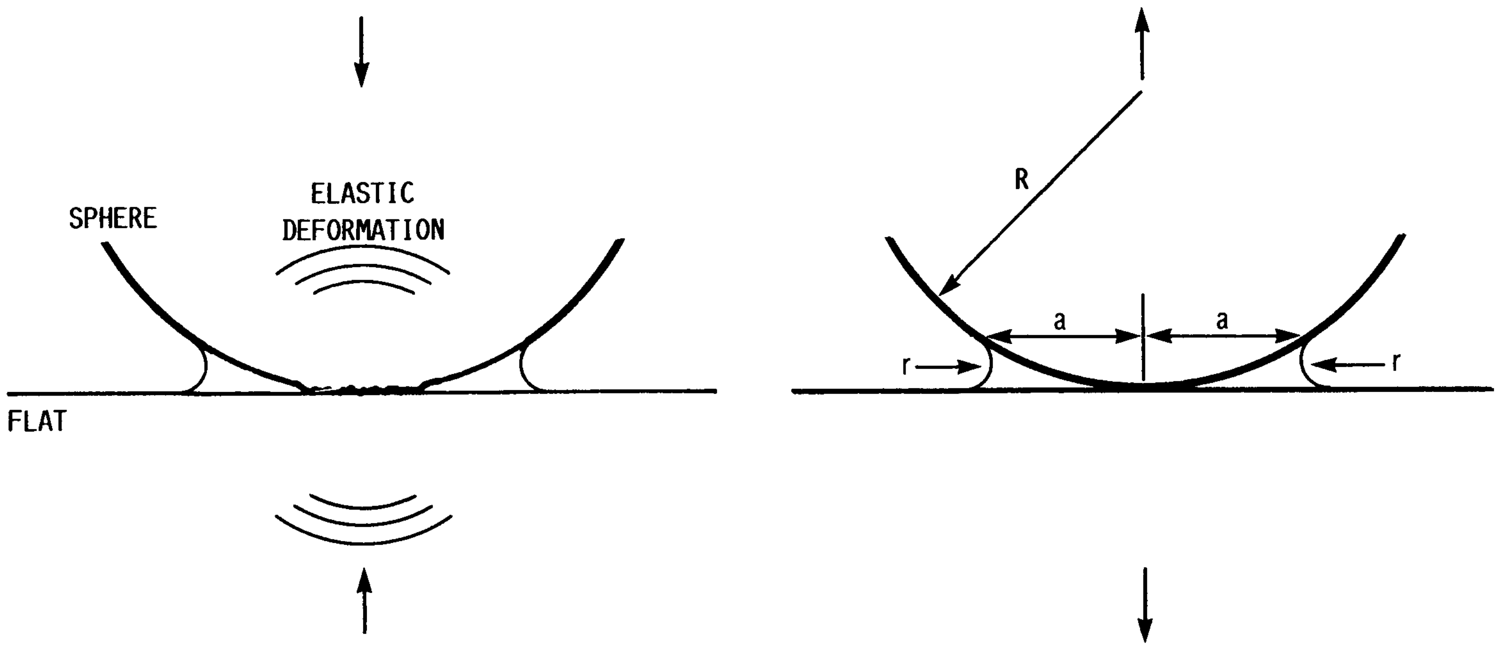


FIGURE 9. - ADHESION AS A FUNCTION OF HUMIDITY FOR A HEMISPHERICAL SILICON NITRIDE PIN IN CONTACT WITH A SILICON NITRIDE FLAT IN DRY, MOIST, AND SATURATED NITROGEN ATMOSPHERE.



(A) CONTACT.

(B) SEPARATION.

FIGURE 10. - MENISCUS FORMED AT CONTACT AREA BETWEEN A SPHERICAL SURFACE AND A FLAT SURFACE.

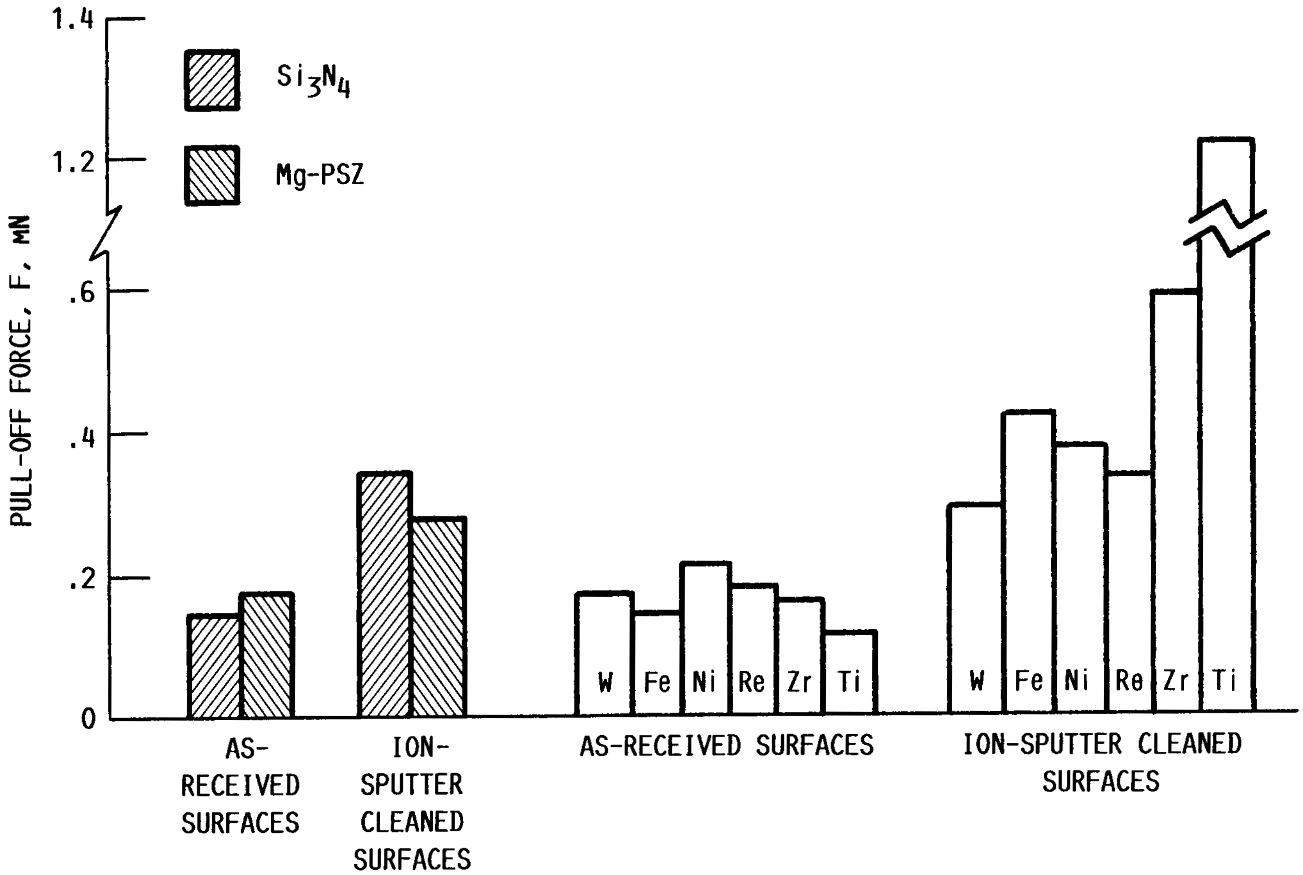


FIGURE 11. - ADHESION FOR AS-RECEIVED AND ION-SPUTTER CLEANED CERAMIC-Si_3N_4 AND METAL-Si_3N_4 INTERFACES. LOAD, 2 mN; VACUUM, 30 nPa; ROOM TEMPERATURE.

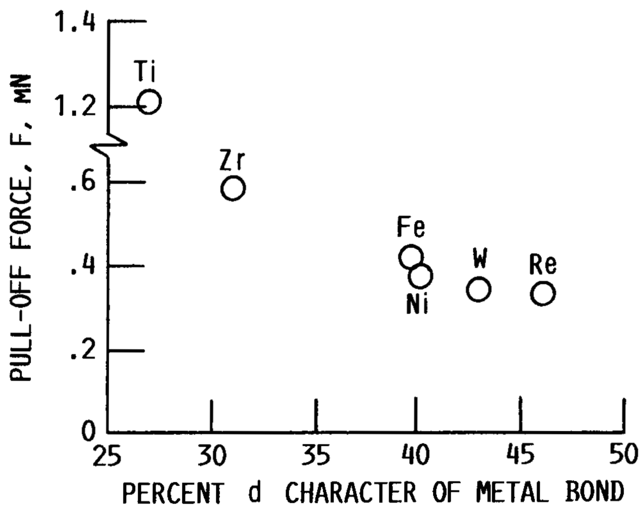


FIGURE 12. - PULL-OFF FORCE (ADHESION) AS A FUNCTION OF THE PERCENTAGE d-BOND CHARACTER OF METALS IN CONTACT WITH SILICON NITRIDE IN VACUUM.

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15. Supplementary Notes

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16. Abstract

A new design of a torsion balance for study of adhesion of ceramics is discussed. A torsion wire and a linear variable differential transformer are used as the sensing mechanism to monitor load and to measure pull-off force (adhesion force). The results of the investigation suggest that this torsion balance has proven to be valuable in studying the interfacial properties of ceramics in controlled environments such as in ultra high vacuum. The pull-off forces measured in dry, moist, and saturated nitrogen atmosphere demonstrate that the adhesion of silicon nitride to silicon nitride contacts remains low at humidities below 80 percent but rises rapidly above 80 percent. The adhesion at saturation is 10 times or more greater than that below 80 percent relative humidity. The adhesion in a saturated atmosphere arises primarily from the surface tension effects of a thin film of water adsorbed on the surfaces. The surface tension of the water film was calculated as 58×10^{-5} to 65×10^{-5} N/cm; the accepted value for water is 72.7×10^{-5} N/cm. Adhesion characteristics of silicon nitride in contact with metals, like the friction characteristics of silicon carbide to metal contacts, can be related to the relative chemical activity of metals in ultra high vacuum. The more active the metal, the higher is the adhesion.

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